TLC, Volatility, and Heat of Solution

STOCKROOM

For part A you will need two TLC developing chambers and two TLC plates (4cm x 10cm each). For part B you will need four Petri dishes with covers, three 50 mL Erlenmeyer flasks, some parafilm, and a 5 mL volumetric pipet. For part C you will need a LabQuest with a temperature probe, a Surface Pro, a calorimeter, a 50 mL Erlenmeyer flask, and two hot plates (one to stir and one to heat).

CHEMICALS

For part A you will need solutions of the following solutes dissolved in dichloromethane: benzoic acid, ethyl benzoate, benzyl alcohol, benzylamine. For part B you will need methanol and ethylene glycol. For part C you will need ammonium nitrate.

OTHER EQUIPMENT

A pencil. Some sort of timer (the clock, your watch, your phone etc.) A 600 mL beaker.

WASTE DISPOSAL

Any solution containing ethylene glycol, dichloromethane, or methanol should go in the organic waste container in the hood.

SAFETY

Wear your goggles the entire time. Be cautious when using the hot plates so as you do not get burned. If you get any chemicals on your skin wash it off immediately with soap and water. The organic solvents used are flammable, keep then away from open flames and sparks. Do not point the UV light at anyone’s eyes, including your own. Perform the TLC portion of the experiment in the hoods.

PROCEDURE

PART A (TLC)

Draw a straight with pencil about 1 cm from the bottom of each TLC plate. Make 4 small marks on that line, also with pencil, equidistant from each other. Do not come within 0.5 cm of either edge of the TLC plate with the outside marks. Label each mark, on each TLC plate, from left to right as 1, 2, 3, and 4. Using a glass capillary TLC spotter spot the marks with the solutions as shown in the table below:

MARK #	SOLUTION
1	Benzoic acid/dichloromethane
2	Ethyl benzoate/dichloromethane
3	Benzyl alcohol/dichloromethane
4	Benzylamine/dichloromethane

Add 10 mL of 10% methanol in dichloromethane to one developing chamber and 10 mL of pure dichloromethane in the other developing chamber.

Take 2 pieces of filter paper and cut a little off of the edges. Roll the filter paper up and put one in each developing chamber, making sure the filter paper touches the bottom of the chamber.

Place one TLC plate in each chamber and close the lid. Allow the eluent to travel up the TLC plate until it is about 0.5 cm from the top.

DO NOT LET THE SOLVENT FRONT REACH THE TOP OF THE TLC PLATE!

Remove the TLC plates from the chambers and **immediately mark the top of the solvent front with a pencil**.

Place each TLC plate under a UV light and outline each observed spot with a pencil.

For both TLC plates measure the distance from the bottom line to the top of the solvent front in centimeters. Record these in your data table to 2 places past the decimal.

Measure the distance from the bottom line to the center of each spot in centimeters for both TLC plates. Record these measurements in your data table to 2 places past the decimal.

PART B (VOLATILITY OF A SOLUTION WITH A NONVOLATILE SOLUTE)

Label each 50 mL Erlenmeyer flask as 25%, 50%, or 75%.

Using the graduated cylinders that are by the reagent bottles, add about 2.50 mL of methanol to the flask labeled 25%, then add about 7.50 mL of ethylene glycol and cover **immediately**.

Record the volume of methanol and the volume of ethylene glycol added to 2 places past the decimal in your data table.

Next add about 5.00 mL of methanol and about 5.00 mL of ethylene glycol to the flask labeled 50% and cover **immediately**.

Record the volume of methanol and the volume of ethylene glycol added to 2 places past the decimal in your data table.

Then, add about 7.50 mL of methanol and about 2.50 mL of ethylene glycol to the flask labeled 75% and cover it **immediately**.

Record the volume of methanol and the volume of ethylene glycol added to 2 places past the decimal in your data table.

Label each petri dish as A, B, C, or D.

Weigh each petri dish with its top and enter the masses in your data table.

Add 5.00 mL of methanol with a volumetric pipet to petri dish A and

immediately cover it with the top.

Add 5.00 mL of the solution from the flask labeled 25% with a volumetric pipet to petri dish B and cover it **immediately.**

Add 5.00 mL of the solution from the flask labeled 50% with a volumetric pipet to petri dish C and cover it **immediately.**

Add 5.00 mL of the solution labeled 75% with a volumetric pipet to petri dish D and cover it **immediately**.

Weigh each petri dish with the solution and cover.

Enter the masses in your data table.

Bring the petri dishes back to your lab bench and uncover all four dishes at the same time and immediately start measuring the time elapsed. After 20 minutes has elapsed cover all four petri dishes.

Reweigh each of the four petri dishes with the cover and remaining solution. Enter the masses in your data table.

PART C (HEAT OF SOLUTION)

CALORIMETER CONSTANT – PART 1

Place the 50 mL Erlenmeyer flask on a balance and tare the balance. Add about 30 mL of D.I. water to the Erlenmeyer flask and record the mass of the water to three places past the decimal in your data table. This is $\mathbf{m_{hot}}$ for part 1.

Place the Erlenmeyer flask on a hot plate and heat until the temperature reaches 95^{o}C or higher.

DO NOT ALLOW THE WATER TO BOIL VERY LONG OR YOU WILL LOSE TOO MUCH MASS!

Set up the LabQuest and SurfacePro. Connect the temperature probe to the

LabQuest. Open the program LoggerPro on the SurfacePro. Click on Experiment → Data Collection and set Length to 300 seconds. Click OK.

Take 2 Styrofoam cups and put one inside the other. Place the cups on a balance and tare the balance.

Add about 70 mL of D.I. water to the cups and record the mass of the cold water to three places past the decimal in your data table. This is $\mathbf{m_{cold}}$ for part 1.

Place the magnetic stir bar into the water, being careful not to splash any water out. Put the plastic lids on top of the cups.

Turn on the stirrer.

Place the temperature probe into the water in your calorimeter.

Record the temperature of the water in the Styrofoam cups in your data table. This is $\mathbf{T_i}$**(cold)** for part 1.

When the water in your Erlenmeyer flask is hot enough, pick up the Erlenmeyer flask with your test tube holder and measure the temperature of the water in the Erlenmeyer flask with the temperature probe as you are adding the hot water to the calorimeter.

Start collecting data with LoggerPro immediately.

Place the lid on the calorimeter.

Record the temperature of the water in the Erlenmeyer flask in your data table. This is $\mathbf{T_i}$**(hot)** for part 1.

In LoggerPro click and drag to highlight the linear portion of your graph.

Click on Analyze → Linear Fit. Record the y-intercept from the box that shows up. This is $\mathbf{T_F}$ for the hot water, the cold water, and the calorimeter.

Empty the cups and dry completely with a paper towel.

HEAT OF SOLUTION Place the dry coffee cups on the balance and tare the balance. Add about 100 mL of D.I. water to the cups and reweigh, recording the mass of the water in your data table.

Add the stir bar and lids to the calorimeter and place it on the stir plate. Place the temperature probe attached to the LabQuest into the water in the calorimeter.

Record the temperature of the water in your date table. This is **T_i(water)** and **T_i(cal)**.

Weigh out between 4 and 4.5 grams of ammonium nitrate. Record the mass to three places past the decimal in your data table.

Add the ammonium nitrate to the calorimeter and start collecting data with the LabQuest. Make sure LoggerPro is still set to collect for 300 seconds.

In LoggerPro click and drag to highlight the linear portion of your graph.

Click on Analyze → Linear Fit. Record the y-intercept from the box that shows up. This is **T_F** for the water and the calorimeter.

<u>CALCULATIONS</u>

PART A

Calculate the R_F for each spot on the TLC plates.

$$R_F = \frac{\text{Distance to middle of spot}}{\text{Distance to solvent front}}$$

R_F is a number (no units) between 0 and 1.

PART B

Calculate the mole fraction of methanol in the 25%, 50%, and 75% solutions. Take the density of methanol as 0.7918 g/mL and the density of ethylene glycol as 1.1132 g/mL.

The molecular formula of methanol is CH_3OH and that of ethylene glycol $C_2H_6O_2$.

Calculate the mass percent of sample remaining for each of the four solutions at the end.

Using Calc, Excel, or another spreadsheet make a graph of mass percent of the sample left after evaporation (y-axis) vs. mole fraction of methanol in the original solution (x-axis). Include a fifth point for pure ethylene glycol (mole fraction methanol = 0.00, percent original sample remaining =100.0). Make a scatter plot and have your spreadsheet draw the best-fit straight line.

PART C

Use your graphs to find $\mathbf{T_F}$ for both parts of part C. Attach all graphs to your lab report.

Calculate your calorimeter constant, **C**. Here is a video explaining these calculations.

Calculate the heat of solution of ammonium nitrate in kJ/mol. To find the energy absorbed or released by the reaction (q_{rxn}) use the energy balance equation below:

$$-q_{rxn} = q_{water} + q_{calorimeter}$$

or

$$-q_{rxn} = m_{water} \cdot s_{water} \cdot \Delta T_{water} + C_{calorimeter} \cdot \Delta T_{calorimeter}$$

Here m_{water} is the mass of water in the calorimeter, s_{water} is 4.184 J/g·°C (this goes in your table of constants), ΔT_{water} is T_f-T_i(water), $C_{calorimeter}$ is the calorimeter constant you found in the first part of part C and $\Delta T_{calorimeter}$ is the same as ΔT_{water}.

To find the heat of solution (ΔH_{soln}) convert q_{rxn} from joules to kilojoules and then divide by the number of moles of ammonium nitrate you weighed out (you will need the molar mass of ammonium nitrate in your table of constants).

Calculate your percent error. Take the true value of the heat of solution as 25.690 kJ/mol.

CONCLUSION

PART A

Report the R_F values you calculated for all 8 spots.

Silica gel has hydroxyl groups on it's surface. The more hydrogen bonds a molecule can form with the silica gel the more tightly it is held, and the smaller it's R_F will be. Look up the structure of the four molecules used: benzoic acid, benzyl alcohol, ethyl benzoate, and benzylamine. Explain the order of elution you got (the relative R_fs).

Methanol will bind to the hydroxyl groups on the silical gel, leaving fewer available to hydrogen bond with the molecules in your spots. Based on this fact, explain the difference between your R_fs for pure dichloromethane and the 10% methanol in dichloromethane.

PART B

Explain the change in mass percent of solution remaining as the mole fraction of methanol changes. Treat the ethylene glycol as a nonvolatile substance. You graph should be close to linear. Why is this? (Hint, think about Rault's law). What could cause deviation from linearity in your graph? (Think about what causes deviations from Raoult's law).

PART C

Report your calorimeter constant, heat of solution in kJ/mol, and percent error. Discuss (thoroughly) at least one possible source of error. Make sure to explain what effect, and why that error would have that effect, for each source of error.